

PATENT SPECIFICATION

(11) 1450271

1450271

- (21) Application No. 377/74 (22) Filed 4 Jan. 1974
- (44) Complete Specification published 22 Sept. 1976
- (51) INT CL² C01B 31/06
- (52) Index at acceptance

C1A J2



(19)

(54) METHOD FOR PRODUCING POLYCRYSTALLINE DIAMONDS AND PREPARATION OF SAME

(71) We, VSESUZHNYY NAUCHNO-ISSLEDOVATELSKY I PROEKTNY INSTITUT TUGOPLAVKIKH METALLOV I TVERDYKH SPLAVOV, a State Enterprise organised and existing under the laws of U.S.S.R., of 56 Shosse Vavshavskoe, Moscow, U.S.S.R., do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the production of diamonds and more particularly to methods for producing polycrystalline diamonds.

Lately a technique for producing diamonds in general and polycrystalline diamonds in particular has been developed in the Soviet Union and in other countries.

This method of producing polycrystalline diamonds involves exposing diamond powders to the effect of a static pressure and high temperatures with or without the use of different additives.

In this case the pressure did not exceed 110 kbar and a temperature of up to 2000°C was used.

As the additives, use was made of such substances as boron, silicon and beryllium which acted as catalysts contributing to better sintering of diamond particles in a polycrystalline monolith.

The main disadvantage of polycrystalline diamonds produced by conventional methods lies in their non-uniform properties attributable apparently to the presence of interlayers of graphite and metallic catalysts in the structure of polycrystals.

The main object of the invention is the provision of a method ensuring optimum conditions for processing diamond powders.

According to the present invention there is provided a method for producing polycrystalline diamonds comprising subjecting a diamond powder located within a high-pressure chamber to the effect of a static pressure at which transformation of graphite into diamond occurs, and a temperature of 2700—

3700°C said diamond powder being in contact with an alkaline earth metal compound.

The most preferred practice is to maintain a temperature within the pressure chamber at from 3100 to 3700°C. In practice it is convenient to maintain a pressure of 120—170 kbar in the pressure chamber.

The alkaline earth metal compound which is introduced into the diamond powders enhances the wear resistance of the polycrystalline diamond.

The alkaline earth metal compound may be fluorspar, limestone, chalk or talc individually or in admixture. It is preferred that the alkaline earth metal compound be introduced when charging the diamond powders into the pressure chamber in the form of layers on top of and beneath the layer of the diamond powder.

In order to produce material suitable for boring tools featuring higher performance characteristics, it is expedient that a mixture of two diamond powders be used with grains of one grade amounting to 0.1 to 2.0 micron in size and of the other one to 5 to 60 microns.

In practice the weight of the diamond powder of larger grain size amounts to 20 to 80% of the total weight of the diamond powders.

It is most preferred that the diamond powder with the larger grain size be equal to 50% of the total weight of both grades.

As a result, the polycrystalline diamond which is obtained which is composed of two kinds of polycrystals, one with structural grains of from 0.1 to 2.0 micron size and one of 5—60 microns size.

Prior to mixing the two sizes of diamond powders it is preferable that the diamond powder with a smaller grain size be subjected to aggregation to form a granular material.

This results in the production of a diamond with polycrystals featuring smaller structural grains dispersed in the body of a polycrystal with larger structural grains.

Characteristic of such material is an enhanced abrasion resistance; it is also not

50

55

60

65

70

75

80

85

90

95

liable to become smooth in service, its surface always remaining rough.

For a better understanding of the invention given hereinbelow is a detailed description of exemplary embodiments thereof, with illustrative examples of diamond materials and methods of their production.

The herein-proposed method envisages the production of polycrystalline diamonds from diamond powders of either a uniform grain size or a mixture of different grain sizes.

The size of the powder particles is not limited, though on certain occasions with a view to obtaining diamonds with specific characteristics it is expedient to use powders with particles of certain predetermined size. Such occasions will be considered in details hereinbelow.

Use can be made of the powders of both natural and artificial diamonds. It is desirable that the powders be subjected to preliminary calcination at a temperature of 500—600°C in an oxidizing atmosphere for 10—45 min. (the duration of the above operation being dependent on the amount of the initial powder, which removes adsorbed gases as well as organic admixtures from the powders. The calcined diamond powders are then charged into a graphite heater and compacted. The graphite heater (which is not described here in detail since it is well known) is placed into a high-pressure chamber. The latter is also commonly known (cf. French Patent No. 2,122,772). In the above chamber the diamond powder is exposed to the effect of a static pressure ranging from 120 to 170 kbar which in practice corresponds to that at which transformation of graphite into diamond takes place. The temperature in this case is maintained within 2700—3700°C. The process proceeds most dependably at a temperature ranging from 3100 to 3700°C.

As a result of the above treatment which lasts 0.7—2.0 sec. the diamond powder is converted into a compact polycrystalline diamond similar in shape to the interior of the graphite heater.

The diamonds obtained are dark grey with a metallic luster and weigh up to 0.5 carats.

A specific gravity of such diamonds is 3.4 to 3.5 g/cm³, uniaxial compression strength is 120 kg/mm². The diamonds have successfully passed tests in drawing wire produced from high-melting metals and copper-nickel alloys and in boring hard and abrasive rocks.

To obtain diamonds with higher strength, a mixture of diamond powders should be used with grain size of from 0.1 to 2.0 micron and from 5 to 60 microns.

In this case the diamond powder with larger grains should amount to 20—80% of the total weight of the diamond powders.

Most favourable strength characteristics are

obtained when the weight ratio of the diamond powders in the above mixture is 50:50.

The above-described blending of the diamond powders and their subsequent processing under the combined effect of high pressures and temperatures results, as stated above, in the production of a diamond composed of two grades of intergrowing polycrystals having accordingly structural grains of different size.

Such diamonds have high strength and abrasive resistance and are intended for use as cutting elements in boring and grinding tools.

If the powder with grains of smaller size is aggregated to form granules prior to mixing, the diamond resulting from the powders processed by the above method is a substance in which the polycrystals with structural grains of smaller size are dispersed in the body of the polycrystal with structural grains of larger size.

The above diamonds are designed for use in grinding and boring tools.

With the above method the powders may be aggregated into granules by the conventional technique. If an adhesive binding agent is used, to form the granules it should be removed by calcination prior to the exposure of the powders to high pressures and temperatures to form the polycrystalline diamond.

When producing diamonds by the method of the invention, it is expedient that the alkaline earth metal compound, such as fluor spar, limestone, chalk, talc, be used either individually or in any desired combination. The above compounds are introduced when the diamond powder is charged into a mould, particularly when placing it in the graphite heater. They are preferably arranged in layers beneath and on top of the layer of diamond powder or the mixture of diamond powders. Although the amount of the compounds is substantial, they are consumed in only negligible amounts, most being later recoverable. The alkaline earth metal compound ensures the production of polycrystalline diamonds with uniform mechanical properties.

Given below are illustrative examples of the diamonds and accordingly the specific features of the production processes.

Example 1

A polycrystalline diamond comprising 70% by wt. of diamond crystals with grains 14 microns in size and 30% by wt. of crystals with grains of 1 micron in size

The diamond was produced from a mixture of diamond powders composed of 70% by weight of the particles ranging in size from 10 to 14 microns and 30% by weight of diamond particles, 1—2 microns in size.

- The diamond powder was charged in a mould in layers with powdered limestone placed on top and beneath the diamond layer and with the amount of limestone being equal to 1/10 of the weight of the diamond powder. The mixture was processed at a pressure of 160 kbar and a temperature of about 3500°C for 0.8 sec.
- Diamond characteristics:
- 10 Colour in cleavage plane —light-grey
Specific gravity —3.42 g/cm³
- Average breaking load per grain 630 microns in size amounted to 19.2 kgf. The diamond is not electrically conducting.
- 15 Example 2
A polycrystalline diamond comprising 50% by wt. of crystals with grains 0.1—1 micron in size and 50% by wt. of crystals with grains ranging in size from 5 to 7 microns
- For obtaining the above polycrystalline diamond a mixture of diamond powders was taken composed of 50% by wt. of a diamond with crystals 0.1—1 micron in size and of 25 50% by wt. of diamonds with crystals 5—7 microns in size.
- The mixture was processed at a pressure of 170 kbar and a temperature of 3400°C for 1.6 sec.
- 30 On top of and beneath the diamond powder a batch of talc was placed amounting to 1/10 of the weight of the diamond powder.
- Diamond characteristics:
- 35 Colour in cleavage plane —light-grey
Specific gravity —3.40 g/cm³
Average breaking load per grain 630 microns in size —21 kgf.
- The diamond is not electrically conducting. It is expedient that such polycrystals be used for tools requiring enhanced strength, such as burnishers.
- Example 3
A polycrystalline diamond comprising 50% of polycrystals with structural grains 0.1—1 micron in size and 50% of diamond grains ranging in size from 10 to 14 microns
- For obtaining the above polycrystalline diamond a mixture of diamond powders was taken comprising 50% by weight of granules 0.3—0.4 mm. in size produced by aggregation of diamond powder with grains 0.1—1 micron in size, and 50% by weight of diamond grains 10—14 microns in size.
- On top and beneath the diamond powder a batch of chalk was placed amounting to 1/10 of the weight of diamond powder.
- Processing was effected at a pressure of 160 kbar and a temperature of 3600°C for 1.8 sec.
- Diamond characteristics:
- Colour in cleavage plane —light-grey
Specific gravity —3.47 g/cm³
- Average breaking load per grain 630 microns in size amounted to 17.5 kgf.
- The diamond is not electrically conducting.
- It is expedient that the diamonds be used in grinding and boring tools since they are not liable to be glazed in service.
- WHAT WE CLAIM IS:—
1. A method for producing polycrystalline diamonds comprising subjecting a diamond powder located within a high-pressure chamber to the effect of a static pressure at which transformation of graphite into diamond occurs, and at a temperature of 2700—3700°C, said diamond powder being in contact with an alkaline earth metal compound.
2. A method as claimed in Claim 1, wherein a temperature of 3100—3700°C is maintained.
3. A method as claimed in Claim 1, wherein a pressure in the range of from 120 to 170 kbar is maintained within the pressure chamber.
4. A method as claimed in Claim 1 or 2 or 3, wherein the alkaline earth metal is fluorspar, limestone, chalk or talc or admixtures thereof.
5. A method as claimed in any preceding Claim, wherein the alkaline earth metal compound forms a layer in contact with a layer of the diamond powder, a layer of said compound being present both above and beneath the layer of the diamond powder.
6. A method as claimed in any preceding Claim, wherein the diamond powder is a mixture of two diamond powders of different grain size, a first powder having a grain size of from 0.1 to 2.0 microns and a second of from 5 to 60 microns.
7. A method as claimed in Claim 6, wherein the said second diamond powder provides from 20 to 80% of the total weight of the powders.
8. A method as claimed in Claim 7, wherein the said second diamond powder provides

50% of the total weight of the diamond powders.

5 9. A method as claimed in Claim 6 or 7 or 8, wherein prior to mixing the said first and second diamond powders the said first diamond powder is formed into aggregate granules.

10 10. A method of producing polycrystalline diamonds substantially as hereinbefore described in the Exemplary embodiments.

11. A polycrystalline diamond whenever produced by the method claimed in any one of Claims 1 to 10.

FITZPATRICKS,
Chartered Patent Agents,
14-18 Cadogan Street,
Glasgow, G2 6QW,
and
Warwick House,
Warwick Court,
London, WC1R 5DJ.

Reference has been directed in pursuance of section 9, subsection (1) of the Patents Act 1949, to Patent No. 1,298,565.

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1976.
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from
which copies may be obtained.